Tuesday, May 12 Facility-specific Workshops

APS Workshop 3

Developing Synchrotron Sample Environments to Study Next-generation Field-driven Device Physics

Location: Bldg. 401, Room A1100

Organizers: Philip Ryan (APS), Markys Cain (National Physical Laboratory, UK), and Paul Thompson (XMaS,

European Synchrotron, France)

Condensed matter physics dominates our technological and economic trajectory, and new exotic phenomena are always considered in the light of applications. Strongly correlated electron physics is a particularly exciting subfield that promises many device variations. It relates to a large class of materials that show extraordinary coupling between several degrees of freedom, including electronic, structure, and magnetic orders. In fact, we are witnessing the emergence of novel device scenarios such as piezotransistors [1], magnetoelectronics [2,3], and mottronics [4], among others.

Microscopically, charge ties many of these behaviors together through piezoelectricity, magneto-electricity, magneto-caloric effects, pyro-electric effects, or multiferroicity. Macroscopically, strain through varying strictive forces acting over longer length scales linearly couples these order parameters, generating exciting possibilities for the innovative device engineer. Use of state-of-the-art synchrotron capabilities has already generated a deep understanding of these coupling properties, and as we develop ways to control these behaviors (either directly through applied fields or physically, i.e., through piezo-strain) we inadvertently generate a new paradigm of physical parameters to be explored. In addition, as we prepare the next generation of smaller and more coherent x-ray synchrotron sources, we are compelled to consider how to take advantage of these properties in order to probe and ultimately control such intricate and powerful phenomena.

It is the goal of this workshop to bring together experts with a strong emphasis on exciting topics regarding emerging device physics and additionally examples of more fundamental topics of strongly correlated phenomena. The resulting discussions will help to guide the APS to envision cutting-edge sample environments and supplemental *in situ* (x-ray) measurements that will augment experimental data and enrich our understanding of the underlying physics.

References

- [1] National Physical Laboratory, "New global research effort to measure nanoscale strain," November 6, 2013, http://www.npl.co.uk/news/new-global-research-effort-to-measure-nanoscal....
- [2] The Piezo Institute, "National Physical Laboratory: Metrology for Advanced Sensors through Engineered Magnetoelectric Coupling," http://www.piezoinstitute.com/about/current-research/metrology-for-advan....
- [3] J.T. Heron et al., "Deterministic switching of ferromagnetism at room temperature using an electric field," *Nature* **516**, 370–373 (2014), doi:10.1038/nature14004.
- [4] H. Yamada et al., "Ferroelectric Control of a Mott Insulator," Scientific Reports 3, 2834 (2013), doi:10.1038/srep02834.

Tuesday, May 12 (all day)

8:30 – 9:00 Philip Ryan (Argonne National Laboratory)

Introductory Remarks





9:00 – 9:40	Markys Cain (National Physical Laboratory, UK) "Nanostrain Project" — Novel Electronic Devices Based on Control of Strain at the Nanoscale
9:40 – 10:00	Glenn Martyna (IBM T.J. Watson Research Center) The Piezoelectronic Transistor: A Stress-driven Next-generation Transduction Device
10:00 – 10:35	Break
10:35 – 11:15	Paul Evans (University of Wisconsin) Nanosecond Electric-field-driven Structural Phase Transitions in Complex Oxides
11:15 – 11:55	Jacob Jones (North Carolina State University) New Direct Measurements of Polarizability Mechanisms in Ferroelectrics via Diffraction and Scattering
11:55 – 1:30	Lunch
1:30 – 2:10	Siddharth Saxena (University of Cambridge, UK) Searching for Multivariable Quantum Criticality
2:10 – 2:50	Peter Fischer (Center for X-ray Optics, Lawrence Berkeley National Laboratory) Magnetic Soft X-ray Spectro-microscopy: Seeing Nanoscale Magnetism in Action
2:50 – 3:20	Break
3:20 – 4:00	Xavi Marti (Institute of Physics, Czech Republic) Antiferromagnetism: Applications of Invisible Magnets
4:00	Sae Hwan Chun (Materials Science Division, Argonne National Laboratory) Static and Dynamic Magnetoelectric Effects in Multiferroic Hexaferrites
Wednesday, May 13 half-day	
9:00 – 9:40	Martin Holt (Center for Nanoscale Materials, Argonne National Laboratory) Strain Imaging of Nanoscale Semiconductor Heterostructures with X-ray Bragg Projection Ptychography
9:40 – 10:20	Jian Liu (University of California, Berkeley) Toward in situ Control and Probe over Novel Magnetoelectric Effects in Magnetic Thin Films and Heterostructures
10:20 – 11:00	Raegan Johnson (Sandia National Laboratory) In situ <i>Electric Field Measurements of Ferroelectric Domain Wall Motion in PZT Thin Films</i>
11:00 – 11:40	Sean McMitchell (University of Liverpool, UK) Developing Traceable Links between Mesoscopic Strain and Crystallography through in situ Interferometrys
11:40 – 12:20	Yong Choi (X-ray Science Division, Argonne National Laboratory) Interfacial Orbital Modification Probed by Polarization-dependent Anomalous X-ray Reflectivity
12:20	Philip Ryan (Argonne National Laboratory) Wrap-up

Introductory Remarks

Philip Ryan

Advanced Photon Source, X-ray Science Division, Argonne National Laboratory, Argonne, IL 60439

I will present a brief vision of the future experimental environment given the anticipated coherence and spatial dimensions that will come with the APS-U project. The general condensed-matter end-user community may question how this affects their user relationship with the facility. My aim in these introductory remarks is to explore how the facility may assuage concerns of negative impacts and more importantly how we, the beamline scientist and user community, can take advantage of the improvements to the beam characteristics. In fact the net potential benefit to the broader community is quite exciting; however, we need to coordinate our efforts to maximize the optimal utilization of the future facility.

WK3

"Nanostrain Project" — Novel Electronic Devices Based on Control of Strain at the Nanoscale

Markys Cain

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Faster, smaller and more energy efficient computing, based on miniature electronic devices, will benefit almost every industrial sector. Recently, materials such as piezoelectrics have been used to develop miniature electronics by allowing the control of properties at the nanoscale via the application of mechanical strain. Piezoelectric materials are uniquely capable of generating precisely defined strains down to very small length scales and are the technology driver for new types of electronic devices.

Currently, there is no measurement framework or facility for traceable measurement of the electromechanical coupling (shown as strain through application of voltage) in piezoelectric materials down to a size of 1 nm.

This project will develop traceable measurements of strain at lengths down to 1 nm and at high electric fields. These measurements need to be non-destructive and should be able to operate on the commercial scale. The results will help develop computing products based on the principles of functional materials such as piezoelectrics.

In this presentation I will explain the following key objectives of the scientific research project, with an emphasis on the first technical goal:

- ▶ Develop links between traceable mesoscale strain metrology and crystallographic strain via *in situ* interferometry and synchrotron x-ray diffraction. This provides for an assessment of the intrinsic piezo response to its, industrially highly relevant, extrinsic (domain mediated) response.
- ▶ Develop ultra-high spatial resolution (100 nm or less) optical methods of strain measurements using IR-SNOM by utilising the PTB synchrotron radiation facility (MLS) in Berlin as an IR light source.
- ▶ Develop traceable validation of macroscale strain metrology in destructive methods including Transmission Electron Microscopy (TEM) and novel holographic TEM, to map intra-grain residual and active (electric field induced) strains. The uncertainty caused by the additional strain from the preparation of TEM slices will also be investigated.
- ▶ Develop the multiphysics materials modelling to underpin all the experimental activities described above, considering both residual, process-related strains in thin film and nano/micro-scale released structures, and electrically driven strains in active devices.



The Piezoelectronic Transistor: A Stress-driven Next-generation Transduction Device

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We have invented a transduction based post-CMOS device based on a piezoelectrically driven metal insulator transition [1]. An input voltage pulse activates a piezoelectric element (PE) which transduces input voltage into an electro-acoustic pulse that in turn drives an insulator to metal transition (IMT) in a piezoresistive element (PR); the transition effectively transduces the electro-acoustic pulse to voltage. Using the known properties of *bulk* materials, we predict using modeling that the PET achieves multi-GHz clock speeds with voltages as low as 0.1 V and a large On/Off switching ratio ($\approx 10^4$) for digital logic [1]. The PET switch is compatible with CMOS-style logic. At larger scale the PET is predicted to function effectively as a large-area low voltage device for use in sensor applications.

PET device performance is enabled by the properties of two materials, a relaxor piezoelectric for the PE and a rare earth chalcogenide piezoresistor for the PR — provided the materials exhibit bulk properties at the nanoscale. Thus it is critical to investigate materials scaling using a combined theoretical/experimental approach. The development of thin film piezoresistive and piezoelectric materials and patterned structures, and associated characterization tools is presented, along with the theoretical models that yield insight into their behavior [2–4]. Integration of these novel materials into 3 evolutionary generations of PET devices, and device characterization, is given [5] to show that a proof of concept has been achieved.

DARPA Mesodynamic Architectures Program under contract number N66001-11-C-4109.

- [1] "High Response Piezoelectric and Piezoresistive Materials for Fast, Low Voltage Switching: Simulation and Theory of Transduction Physics at the Nanometer-Scale," D.M. Newns, B.G. Elmegreen, X.-H. Liu, and G.J. Martyna, Adv. Mat. 24, 3672 (2012).
- [2] "Giant Piezoresistive On/Off Ratios in Rare-Earth Chalcogenide Thin Films Enabling Nanomechanical Switching," M. Copel, G.J. Martyna, and D.M. Newns et al., Nano Lett. 13, 4650 (2013).
- [3] "Anisotropic strain in SmSe and SmTe: implications for electronic transport," M.A. Kuroda, Z. Jiang, M. Povolotskyi, G. Klimeck, D.M. Newns, and G.J. Martyna, *Phys. Rev. B.* **90**, 245124 (2014).
- [4] "Lateral scaling of PMN-PT thin films for piezoelectric logic," R. Keech, S. Shetty, M.A. Kuroda, X.-H. Liu, G.J. Martyna, D.M. Newns, and S. Trolier-McKinstry, *J. Appl. Phys.* 115, 234106 (2014).
- [5] "Pathway to the Piezo Electronic Transduction Logic Device," accepted NanoLetters (2014).

WK3

Nanosecond Electric-field-driven Structural Phase Transitions in Complex Oxides Paul G. Evans

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Complex oxide electronic materials, including ferroelectric and multiferroics, can exhibit large responses to externally applied fields when the system has a composition or external stresses that place the system near a boundary between structural phases. The development of time-resolved x-ray diffraction and scattering techniques allows us to probe the dynamics and energetics of piezoelectric distortion and phase transformation in complex oxide electronic materials at such boundaries, with both nanosecond time resolution and a high level of structural precision. Compressive epitaxial stresses can place thin films of the multiferroic oxide BiFeO₃ at a boundary between phases with different structural extent. The electric-field driven transition exhibits contributions from both stably switching and reversible components, and can occur at times as short as tens of nanoseconds. Comparison with density functional theory results shows that the transition can be understood by comparing the free energies of the piezoelectrically distorted structures. Control of this transition, and other field-driven phase transition phenomena, has the potential to provide an additional route to the control of electronic and magnetic properties of complex oxides.

New Direct Measurements of Polarizability Mechanisms in Ferroelectrics via Diffraction and Scattering

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The functionality of many dielectric and ferroelectric materials is dependent on the external application of electric fields. Characterizing the response of the material at length scales ranging from sub-nanometer to micrometer is important for understanding and engineering these functionalities. For field-induced strain, for example, both the contribution of the intrinsic piezoelectric effect (originating at sub-nanometer dimensions) and ferroelectric/ferroelastic domain wall motion (at the nanometer length scale) contribute to strain. For both polarizability and permittivity, it is important to characterize and understand the field-induced response of the local structure (i.e., ionic polarizability) and changes in the domain structure (i.e., dipolar). In this talk, we present the new development of two different methods to characterize both local structure and domain structure changes during field application and the contribution of these mechanisms to polarization.

In the first method, we have used x-ray diffraction *in situ* during application of electric fields in BaTiO₃ to determine the contribution of ferroelectric/ferroelastic (i.e., non-180°) domain walls to the polarization. We have used a complementary analysis approach to further determine the contribution from ferroelectric (i.e., 180°) domain walls to the polarization.

In the second method, we have used *in situ* total x-ray scattering during application of electric fields and determined pair distribution functions (PDFs). The field-dependent PDFs enable the assessment of local structure changes in $BaTiO_3$ and $Na_{0.5}Bi_{0.5}TiO_3$. A unique dipolar mechanism is observed at the unit-cell level in $Na_{0.5}Bi_{0.5}TiO_3$ which involves rearrangements of Bi^{3+} displacements. In contrast, $BaTiO_3$ exhibits a smaller local scale response to the electric field which is nonetheless consistent with piezoelectric strain.

The results are interpreted holistically to enable ascription of dominant mechanisms of polarizability in dielectric and ferroelectric materials. For example, the first and second largest contributions to polarizability in BaTiO₃ are 180° domain wall motion and non-180° domain wall motion. The new characterization methods can be readily applied to other materials.

WK3

Searching for Multivariable Quantum Criticality

Siddharth S. Saxena

Cavendish Laboratory, Madingley Road, Cambridge, CB3 0HE, Great Britain

This talk will discuss pressure induced phenomena in the vicinity of magnetic quantum phase transitions and Quantum Criticality in Metallic and Insulating Systems.

Materials tuned to the neighbourhood of a zero temperature phase transition often show the emergence of novel quantum phenomena. Much of the effort to study these new emergent effects, like the breakdown of the conventional Fermi-liquid theory in metals has been focused in narrow band electronic systems. Ferroelectric crystals provide a very different type of quantum criticality that arises purely from the crystalline lattice. In many cases the ferroelectric phase can be tuned to absolute zero using hydrostatic pressure. Close to such a zero temperature phase transition, the dielectric constant and other quantities change into radically unconventional forms due to the fluctuations experienced in this region. The simplest ferroelectrics may form a text-book paradigm of quantum criticality in the



solid-state where there are no complicating effects of electron damping of the quantum charge fluctuations. We present low temperature high precision data demonstrating these effects in pure single crystals of SrTiO₃ and KTaO₃. We outline a model for describing the physics of ferroelectrics close to quantum criticality and highlight the expected 1/T2 dependence of the dielectric constant measured over a wide temperature range at low temperatures. In the neighbourhood of the quantum critical point we report the emergence of a small frequency independent peak in the dielectric constant at approximately 2K in SrTiO₃ and 3K in KTaO₃. Looking to the future, we imagine that quantum paraelectric fluctuations may lead to new low temperature states and mediate novel interactions in multi-ferroic systems (e.g., EuTiO₃) and ferroelectric crystals supporting itinerant electrons.

WK3

Magnetic Soft X-ray Spectro-microscopy: Seeing Nanoscale Magnetism in Action Peter Fischer

Center for X-ray Optics, Lawrence Berkeley National Laboratory, Berkeley, CA 94720 Physics Department, University of California, Santa Cruz, CA 94056

The era of nanomagnetism, which aims to understanding and controlling magnetic properties and behavior on the nanoscale, is currently expanding into the mesoscale [1], which will harness complexity and novel functionalities, which are essential parameters to meet future challenges in terms of speed, size and energy efficiency of spin driven devices. Multimodal characterization techniques, such as tomographic magnetic imaging and investigations of spin dynamics down to fundamental magnetic length and time scales with elemental sensitivity in emerging multi-component materials will enable future scientific breakthroughs. I will review recent developments with full-field magnetic soft x-ray transmission microscopy [2] to study spin configurations in magnetic nanotubes [3], the stochastic behavior in vortices [4] and the local distribution of magnetic properties near domain walls [5].

This work was supported by the Director, Office of Science, Office of Basic Energy Sciences, Materials Sciences and Engineering Division, of the U.S. Department of Energy under Contract No. DE-AC02-05-CH1123 and by the Leading Foreign Research Institute Recruitment Program (Grant No. 2012K1A4A3053565) through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (MEST).

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- [3] M.-Y. Im et al., Nature Communications 5 5620 (2014).
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- [5] M.J. Robertson et al., J Appl. Phys. 117 17D145 (2015).

WK3

Antiferromagnetism: Applications of Invisible Magnets

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In 1970, at the time when compact cassettes made it to the market after a century of experimenting with ferromagnetic storage, the Nobel Prize was awarded for the "fundamental work and discoveries concerning antiferromagnetism." Louis Neel pointed out in his Nobel lecture that while abundant and interesting from a theoretical viewpoint, antiferromagnets did not seem to have any applications. Indeed, the alternating directions of magnetic moments on individual atoms and the resulting zero net magnetization make antiferromagnets hard to control by tools common in ferromagnets. While preoccupied with the inherent difficulties to read and write magnetic information in antiferromagnets, scientists and engineers have largely overlooked the positive sides of antiferromagnets as being magnetically invisible [1] and uniquely robust against magnetic perturbations [2]. In this talk, we will review recent developments in the emerging field of antiferromagnetic spintronics, retracing the footsteps

of the ferromagnetic based technologies. We will examine the path starting from seminal basic science experiments that took place at the Advanced Photon Source down to a survey of the potential markets for the coming up *invisible magnetism*.

- [1] https://www.youtube.com/watch?v=X1Ft_OnRaq4.
- [2] https://www.youtube.com/watch?v=HWZLJ02sb0U.

WK3

Static and Dynamic Magnetoelectric Effects in Multiferroic Hexaferrites Sae Hwan Chun

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Multiferroics, wherein magnetism and ferroelectricity coexist, are of great interest for the prospect of new multifunctional devices by using magnetoelectric (ME) effects through the cross-coupling between the magnetic and electric properties. In most multiferroics currently known, however, controlling electric polarization with magnetic field or magnetization with electric field has been realized only at low temperatures. In addition, their ME susceptibilities are too small for practical applications. Hence, it is essential to improve both the operating temperature and the ME sensitivity of magnetic ferroelectrics for use in ME devices. Investigating the multiferroic hexaferrites, we discovered a novel chemical route effectively tailoring the electric polarization induced by a low magnetic field in (Ba,Sr)₂Zn₂Fe₁₂O₂₂ (Zn₂Y-type) hexaferrite by Al-substitution to possess a giant magnetoelectric susceptibility (MES) [1]. Furthermore, in (Ba,Sr)₃Co₂Fe₂₄O₄₁ (Co₂Z-type) hexaferrite single crystals with large MES, we realized the control of magnetization by an electric field at room temperature [2]. In addition to those static ME properties, a dynamic ME effect, electric-dipole-active magnon resonance in THz frequency range, is found in the Co₂Z-type hexaferrite, exhibiting the spectral weight even at room temperature [3]. The unprecedented supreme static and dynamic ME phenomena in the hexaferrites may provide a pathway to overcome the challenge in application of multiferroics for real devices.

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WK3

Strain Imaging of Nanoscale Semiconductor Heterostructures with X-ray Bragg Projection Ptychography

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We report the imaging of nanoscale strain distributions in complementary components of lithographically engineered epitaxial thin film semiconductor channel heterostructures using synchrotron x-ray Bragg Projection Ptychography (BPP). A new phase analysis technique applied to the reconstructed BPP phase images from two laterally adjacent, stressed materials produced lattice strain and lattice rotation maps with a spatial resolution of ~15 nm, a strain sensitivity of better than 0.01%, and an angular resolution of ~0.1 mrad [1].

Bragg projection ptychography is a coherent diffraction x-ray imaging technique capable of mapping structural perturbation, such as strain, in single crystal thin films with nanoscale spatial resolution [2,3]. In this study, analysis of the orthogonal derivatives of the reconstructed phase maps provides insight into two distinct lattice responses that quantitatively agree with linear elastic predictions. This demonstrates that Bragg ptychography can be used to quantitatively visualize extremely subtle lattice perturbations at the nanoscale under realistic conditions without sectioning or otherwise modifying the boundary conditions of the sample.



- [1] M.V. Holt et al., Phys. Rev. Lett. 112, 165502 (2014).
- [2] S.O. Hruszkewycz et al., Nano Lett. 12, 5148 (2012).
- [3] S.O. Hruszkewycz et al., Phys. Rev. Lett. 110, 177601 (2013).

Toward *in situ* Control and Probe over Novel Magnetoelectric Effects in Magnetic Thin Films and Heterostructures

Jian Liu

Department of Physics, University of California, Berkeley, CA 94720 Materials Science Division, Lawrence Berkeley National Laboratory, Berkeley, CA 94720

Magnetoelectric effects, which couple the spin and charge degree of freedom, are the key to the great success of various magnetic material-based technologies, such as recording and memories. The development of the next generation of magnetic devices with improved performance or new functionalities relies on creating, understanding, and controlling novel magnetoelectric effect/coupling. As a class of materials where the magnetic and electronic properties are tightly related, complex oxides present great potential in this area. The challenge, however, has been imposed by the fact that most magnetic oxides are antiferromagnetic and difficult to harness or probe at nanoscale. In this talk, I will show some of our recent work on different magnetoelectric effects in oxide heterostructures with antiferromagnetism as a key component. In particular, results that utilize synchrotron-based probes will be discussed.

WK3

In situ Electric Field Measurements of Ferroelastic Domain Wall Motion in PZT Thin Films

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Lead zirconate titanate (PZT) thin films are used in microelectromechanical systems (MEMS) due to their large piezoelectric response. In order to quantitatively study this response, *in situ* electric field measurements were performed at the Advanced Photon Source at Argonne National Laboratory. The electromechanical response in PZT is a result of both the intrinsic (lattice) piezoelectric effect as well as the motion of ferroelectric and ferroelastic domain walls (extrinsic effect). In-depth studies of the intrinsic and extrinsic effects in bulk materials has been performed on a number of ceramic materials, however, thin films present a challenge due to the significantly smaller volume of material. This talk will outline the challenges and describe the experimental setup used to perform the *in situ* measurements on PZT thin films. In addition, the work presented will describe direct measurements of ferroelastic domain wall motion in 2-µm thick {001} oriented PZT (30/70) films under different release states. Films that were fully clamped to the underlying substrate are compared with films that are up to 75% released from the wafer. The released films showed significantly larger domain wall motion response based on the changes in peak intensity between the 200 and 002 diffraction peaks. Quantitative details of the influence of the release state and electric field will be discussed.

Developing Traceable Links between Mesoscopic Strain and Crystallography through *in situ* Interferometry

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Recently, there has been considerable research effort on understanding the complex interplay between material structure and the internal strain in piezo and ferroelectrics, and multiferroics. This is a key factor in the functional efficiency of devices.

Of particular relevance is the correlation between strain and electric polarisation, which is being exploiting to develop a novel Piezoelectric-Effect-Transistor (PET), which offers a possible route to replace current CMOS technology. To aid the development of this transformative technology, several European national laboratories, academic and commercial partners formed a consortium funded through the European Metrology Research Programme (EMRP) Project IND54 Nanostrain. The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.

The operation of the PET will be controlled through application of an electric field. It is therefore imperative to investigate the physical deformation and strain state that occurs under applied electric fields *in situ* and *in operando*. We have incorporated a dual-beam optical interferometer onto the XMaS beamline at the ESRF. *In situ* polarisation, lattice parameter, and deformation measurements allow new insights into the correlation between induced strain and material properties in piezoelectrics. We will detail experimental strategies employed to reduce noise and show results from both static (d.c.) and dynamic cycles (up-to 55Hz) of electric field. Standard piezoelectric single crystals and thin films were used to develop quantitative and traceable metrologies for the precise determination of bulk and atomic strain within these structures. The addition of magnetic field and temperature dependence is also discussed.

WK3

Interfacial Orbital Modification Probed by Polarization Dependent Anomalous X-ray Reflectivity

Yong Choi

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In a lattice matched multilayer structure, materials with similar structures but dissimilar properties can be forced to coexist at the interfaces. Numerous recent studies have shown novel interfacial properties that are not intrinsic to either of the constituent materials. We investigate the role of interfacial modification in the metal-to-insulator transition observed from superlattices of two RNiO₃ (R=rare earth) with different transition behaviors, using a combined approach between spectroscopy and scattering to take advantage of the element-specificity of x-ray absorption near edge spectroscopy and the enhanced interfacial sensitivity of x-ray reflectivity.